

ALLOY DESIGN OF TEMPERED MARTENSITIC 9Cr-BORON STEEL FOR A-USC BOILERS

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ABSTRACT

The addition of boron without the formation of any boron nitrides during normalizing heat treatment at high temperature minimizes the degradation in creep strength of both base metal and welded joints of 9Cr steel at 650 °C and long times. The enrichment of soluble boron near prior austenite grain boundaries (PAGBs) by the segregation is essential for the reduction of coarsening rate of $M_{23}C_6$ carbides in the vicinity of PAGBs, enhancing boundary and subboundary hardening, and also for the production of same microstructure between the base metal and heat-affected-zone (HAZ) in welded joints, indicating no Type IV fracture in HAZ. Excess addition of boron and nitrogen promotes the formation of boron nitrides during normalizing, which reduces the soluble boron concentration and accelerates the degradation in creep rupture ductility at long times. 9Cr-3W-3Co-VNb steel with 120 - 150 ppm boron and 60 - 90 ppm nitrogen (MARBN) exhibits not only much higher creep strength of base metal than Gr.92 but also substantially no degradation in creep strength due to Type IV fracture at 650 °C. The pre-oxidation treatment in Ar gas promotes the formation of protective Cr_2O_3 scale on the surface of 9Cr steel, which significantly improves the oxidation resistance in steam at 650 °C.

INTRODUCTION

Advanced ultra-supercritical (A-USC) power plants with steam temperature of 700 °C or above require 9 to 12Cr martensitic steels in order to minimize the requirement of expensive nickel-base superalloys [1]. Nickel-base superalloys are candidates for the highest temperature components of A-USC power plants, while 9 to 12Cr martensitic steels can be used for the next highest temperature components. The maximum allowable temperature of Creep Strength Enhanced Ferritic (CSEF) steels Grades 91, 92 and 122 is 610 to 620 °C. Expansion of the maximum allowable temperature up to 650 °C is highly desirable for next-generation ferritic steels. Critical issues for the development of ferritic steels for 650 °C boilers are the improvement of oxidation resistance as well as long-term creep rupture strength, including welded joints. In particular, the degradation in creep strength due to Type IV fracture in welded joints is serious for CSEF steels at temperatures above 600 °C. The purpose of the present paper is to make clear the alloy design philosophy for 9Cr-boron steel to achieve long-term safety in terms of long-term creep strength, ductility, resistance to Type IV cracking and oxidation resistance at 650 °C, beyond Grades 91, 92 and 122.

EXPERIMENTAL PROCEDURE

0.08C-9Cr-3W-3Co-0.2V-0.05Nb (in nominal) steel with different boron concentrations of 0, 48, 92 and 139 ppm was used to investigate the effect of boron on creep strength of 9Cr boron steel base metal [2]. Nitrogen was not added to the steel to avoid the formation of boron nitrides (BN) during normalizing heat treatment at high temperature. Although no nitrogen was added to the steel, residual nitrogen was analyzed to be 10 to 30 ppm. Residual Al was also as low as 30 to 50 ppm. The 9Cr-3W-3Co-0.2V-0.05Nb steel with 140 ppm boron but different nitrogen concentrations was used to investigate the effect of nitrogen on creep strength of 9Cr-boron steel [3]. The steels were basically prepared by vacuum induction melting to 50 - 100 kg ingots. Creep tests for the base metal were carried out at 650 °C for up to about 9×10^4 h, using specimens of 10 mm in gauge diameter and 50 mm in gauge length. The welded joints were prepared by means of multi-layered Gas Tungsten Arc (GTA) welding [4]. The simulated heat-affected-zone (HAZ) specimens were also prepared.

IMPROVEMENT OF LONG-TERM CREEP STRENGTH OF BASE METAL BY BORON

Figure 1 shows the creep rupture data for 9Cr-3W-3Co-0.2V-0.05Nb steel with different boron concentrations at 650 °C. Nitrogen was not added to the steel. The base steel with no boron (0 ppm boron) exhibits a loss of creep rupture strength at long times above about 1000 h at 650 °C. With increasing boron content, however, the time to rupture significantly increases at low stresses. The creep rupture strength of the present 9Cr steel with 139 ppm boron (residual nitrogen concentration : 34 ppm) is evaluated to be 80 MPa at 650 °C and 10^5 h. The long-term creep rupture strength of the present 9Cr steel with 92 ppm boron is roughly the same as T/P92 [5], while that of the present 9Cr steel with 48 ppm boron is roughly the same as T91 [6].

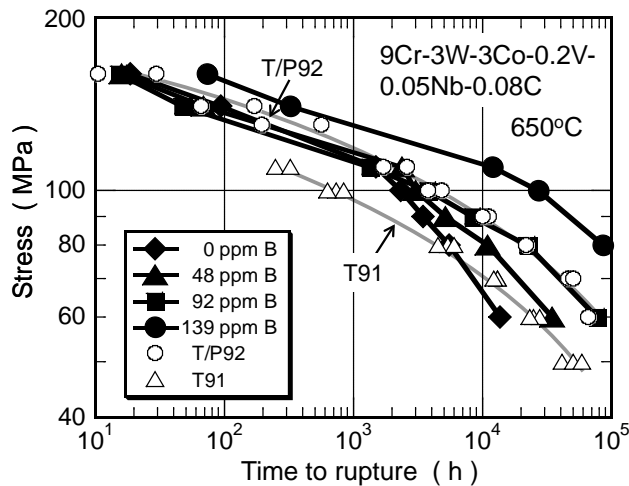


Fig.1 Effect of boron on creep rupture strength of 9Cr-3W-3Co-0.2V-0.05Nb steel at 650 °C, together with data for T/P92 and T91.

The addition of boron also improves the creep rupture ductility at 650 °C and long times as shown in Fig.2 for the reduction of area, although the reduction of area is roughly the same among the steels containing 48 to 139 ppm boron. This suggests that the localization of creep deformation near prior austenite grain boundaries (PAGBs) is less pronounced by the addition of boron. The reduction of area after long-term creep rupture testing is in order of 9Cr-boron steel => T91 > T/P92. The improved reduction of area is advantageous to the creep-fatigue life, because the creep-fatigue life is proportional to the reduction of area in creep rupture test, namely, proportional to the creep ductility but not proportional to the creep strength [7].

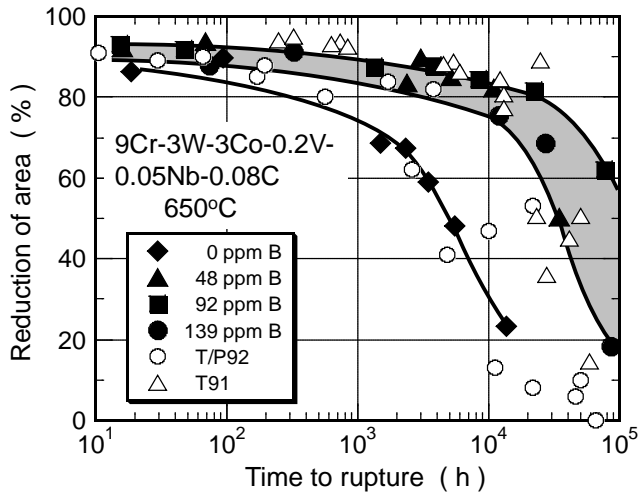


Fig.2 Effect of boron on reduction of area of 9Cr-3W-3Co-0.2V-0.05Nb steel at 650 °C, together with data for T/P92 and T91.

The increase in boron concentration decreases the minimum creep rate. The onset of acceleration creep is retarded and the transient creep region continues for longer times with increasing boron concentration. The minimum creep rate is inversely proportional to the time to minimum creep rate or the duration of transient creep. The longer duration of transient creep results in a lower minimum creep rate and a longer time to rupture. The present author has revealed for tempered martensitic 9Cr steel that the transient creep is basically a consequence of the movement and annihilation of high-density dislocations and that the acceleration creep is a consequence of the gradual loss of creep strength due to microstructure recovery [8]. The onset of acceleration creep is closely correlated with the migration of lath or block boundaries, causing the coarsening of laths or blocks. The time to minimum creep rate, which reflects the microstructure stability during creep, is a deciding parameter for the creep life.

Analysis by scanning Auger spectroscopy for the 9Cr steel with 139 ppm boron after aging at 650 °C for 10,300 h shows that boron is enriched in $M_{23}C_6$ carbides in the vicinity of PAGBs as shown in Fig.3 [2]. The enrichment of boron in $M_{23}C_6$ carbides is also observed after tempering, but it becomes more significant with increasing aging time. No evidence is found for the enrichment of boron in the Fe_2W Laves phase, which precipitated during creep at 650 °C. The addition of boron reduces the rate of Ostwald ripening of $M_{23}C_6$ carbides in the vicinity of PAGBs during creep. Sub-boundary hardening enhanced by fine distributions of $M_{23}C_6$ carbides is the most important factor in creep strengthening at long times. Recently, Liu and co-workers [9] analyzed boron in TAF B steel (10.45Cr-1.54Mo- 0.24V-0.18Nb-0.21C-0.017N-0.03B) by means

of atom-probe tomography (APT) and found that boron was enriched in $M_{23}C_6$ carbides, which was more significant in the vicinity of PAGBs than inside grain, the same as the present results.

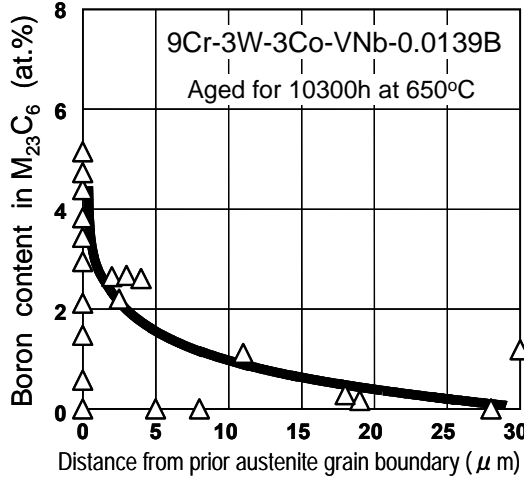


Fig.3 Enrichment of boron in $M_{23}C_6$ carbides near PAGBs in 9Cr-3W-3Co-0.2V-0.05Nb-0.08C steel with 139ppm boron after aging for 10,300 h at 650 °C.

Additive strengthening due to boron and MX carbonitrides

Figure 4(a) shows the effect of nitrogen addition on the time to rupture and minimum creep rate of the 9Cr steel containing about 140 ppm boron at 650 °C and 120 MPa. The peaks of the time to rupture and minimum creep rate are located at 80 to 100 ppm nitrogen, which corresponds to the maximum solid solubility of nitrogen in equilibrium with BN in the steel with 140 ppm boron at a normalizing temperature of 1100 °C, as shown in Fig.4(b). This indicates that the addition of nitrogen without the formation of any BN during normalizing significantly improves the creep strength but excess addition of nitrogen causes the formation of BN during normalizing heat treatment, degrading the creep strength. At a boron concentration of 140 ppm, only 95 ppm nitrogen can dissolve in the matrix at normalizing temperature [10]. Figure 5 shows the

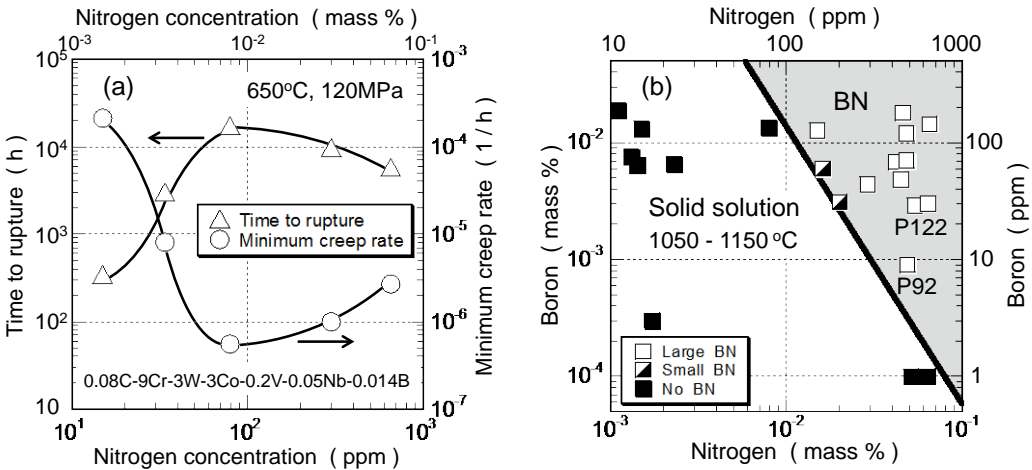


Fig.4 (a) Effect of nitrogen on time to rupture and minimum creep rate of 9Cr steel with 140 ppm boron and (b) composition diagram of boron and nitrogen for 9 to 12Cr steels.

formation of boron nitrides of several μm size in the present steel containing 140 ppm boron and 650 ppm nitrogen and in Gr.92 after normalizing.

Excess addition of boron and nitrogen degrades the creep rupture ductility as well as the creep strength at 650°C and long times, as shown in Fig.6. The degradation in reduction of area is significant in the present steel containing 300 and 650 ppm N, where a large amount of BN formed during normalizing. On the other hand, the present steel containing nitrogen less than 100 ppm exhibits the enough reduction of area, similar as T91.

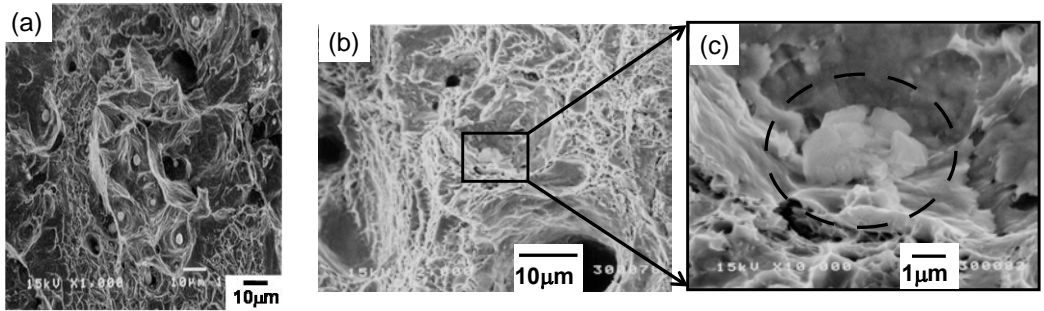


Fig.5 Boron nitrides in (a) present 9Cr steel with 140 ppm boron and 650 ppm nitrogen, (b) and (c) Gr.92, after ductile fracture at room temperature.

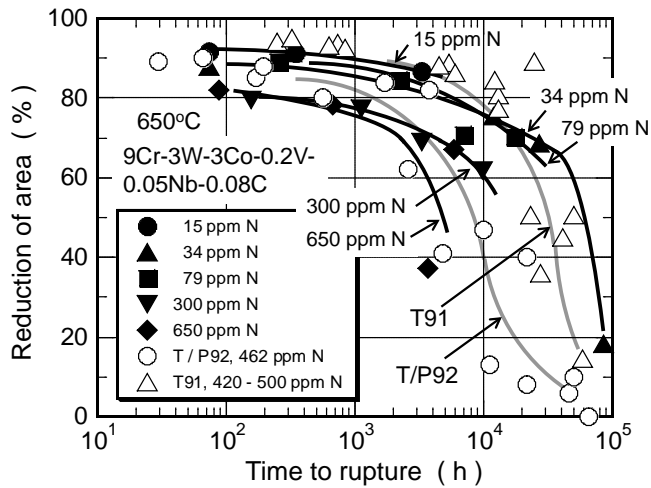


Fig.6 Effect of nitrogen on reduction of area of 9Cr-3W-3Co-0.2V-0.05Nb steel with 140 ppm boron at 650°C , together with data for T/P92 and T91.

The effect of nitrogen addition on the creep deformation behavior is shown in Fig.7(a), where the creep rate versus time curves of the steel at 650°C and 120 MPa are shown. The addition of nitrogen causes a rapid decrease in creep rate with time in the transient region. The creep rates in the transient region are the same among the three steels containing different nitrogen concentrations of 79, 300 and 650 ppm. Based on the results of the steel containing very low nitrogen of 15 ppm, the onset of acceleration creep is retarded up to longer time by the addition of 79 ppm nitrogen but again it shifts to earlier times with increasing nitrogen concentration above 79 ppm. The dissolved nitrogen concentration is roughly the same between the two steels

containing 79 and 650 ppm nitrogen after tempering, Fig.7(b). Dissolved nitrogen can precipitate as fine MX carbonitrides during creep at 650 °C. Indeed, very fine vanadium-rich MX carbonitrides were observed to have precipitated in the steel containing 79 ppm nitrogen during aging for 1000 h at 650 °C [3]. The fine MX carbonitrides precipitated during creep effectively decreases the creep rates in the transient region and also stabilize the martensitic microstructure by retarding dislocation recovery. The difference in onset time of acceleration creep between the two steels containing 15 and 79 ppm nitrogen is due to the microstructure stabilization due to distributions of fine MX carbonitrides. The addition of excess nitrogen of 300 and 650 ppm reduces soluble boron available for microstructure stabilization by the formation of BN during normalizing. This is a reason why the onset of acceleration creep takes place at earlier times in the high nitrogen steels containing 300 and 650 ppm than in the low nitrogen steel containing 79 ppm.

9Cr-3W-3Co-0.2V-0.05Nb steel with 140 ppm B

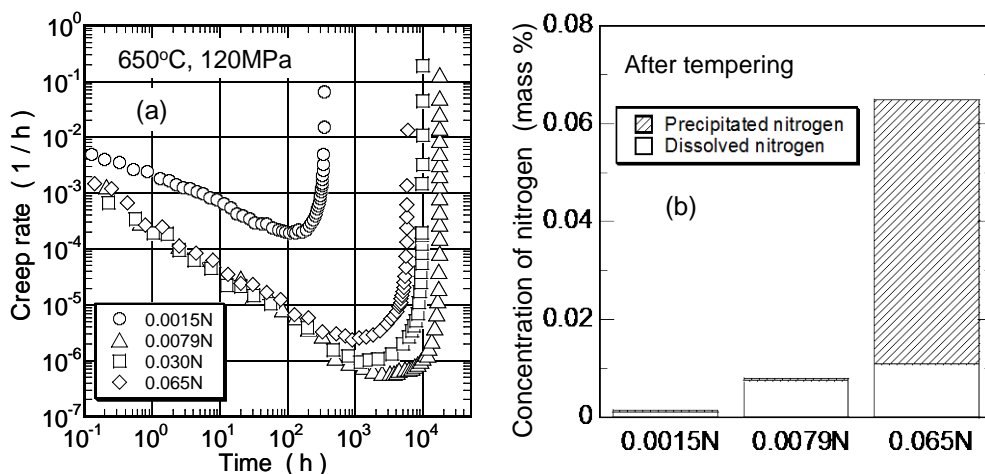


Fig.7 (a) Effect of nitrogen on creep rate versus time curves of the steels and (b) dissolved and precipitated nitrogen concentrations in the steels after tempering.

Suppression of Type IV fracture in welded joints

The addition of boron and nitrogen without any formation of BN during normalizing heat treatment causes no grain refinement and no Type IV fracture in HAZ of 9Cr steel welded joints, Fig.8 [11-12]. Soluble boron is essential also for the suppression of Type IV fracture.

The 9Cr-3W-3Co-0.2V-0.05Nb steel containing 130 ppm boron consists of coarse grains of roughly 200 to 300 μm after the thermal cycle to a peak temperature of 950 °C, just above the A_{C3} temperature, which is substantially the same as that of base metal, as shown in Fig.9 [13], where GBs are traced so that grain size is clearly identified. Fine grains form along GBs of coarse grains but are limited only in the vicinity of GBs. Fe₇W₆ μ phase precipitates at GBs of fine grains during PWHT. Inside the grains, the martensitic microstructure substantially consists of blocks but laths are not clearly seen, Fig.10(a). Not only PAGBs but also block boundaries are covered by enough precipitates, suggesting enough boundary and sub-boundary hardening, substantially the same as the base metal.

The A_{C3} simulated-HAZ specimens of Gr.92 exhibits the fine-grained microstructure, where very few $M_{23}C_6$ carbides are formed along PAGBs, Fig.10(b) [14]. Inside the grains, Gr.92

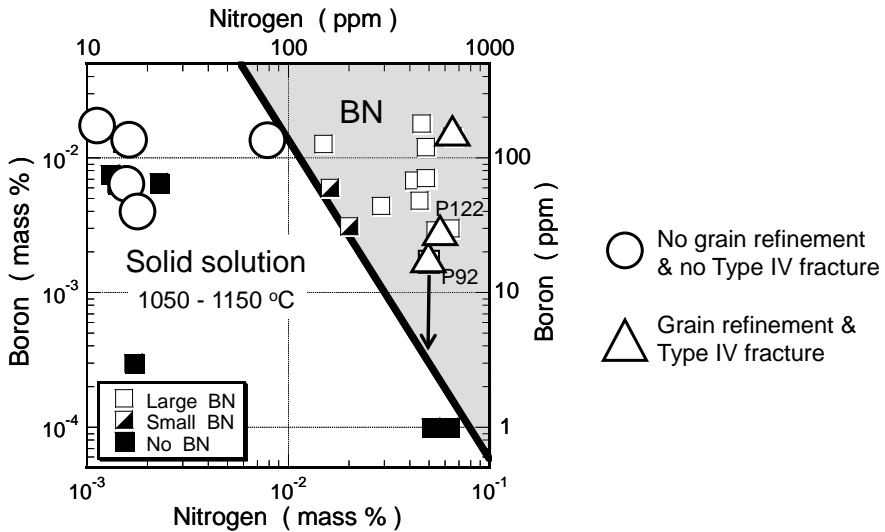


Fig.8 Grain refinement and Type IV fracture in 9 to 12Cr steel welded joints.

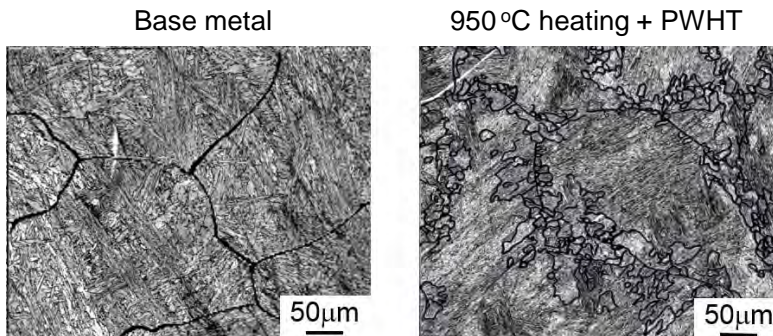


Fig.9 Optical micrographs of 9Cr-boron steel containing 130 ppm boron: base metal and after thermal cycle to a peak temperature of 950°C (near A_{C3}) and subsequent PWHT.

consists of equiaxed subgrains with low density of dislocations and sparse distributions of large carbides. These results suggest that the boundary and subboundary hardening is significantly reduced in Gr.92 after the A_{C3} thermal cycle. The degradation in creep life of Gr.92 after the A_{C3} thermal cycle is not caused by the grain refinement but that the reduction of boundary and subboundary hardening is the most important. Diffusive α/γ transformation takes place in Gr.92 during heating, while at first fine grains form at grain boundaries in the 9Cr-boron steel by diffusive transformation and then martensitic α/γ transformation takes place inside the grain [13].

Using a binding energy of 62.7 kJ mol^{-1} [15] reported for type 316 stainless steel, segregation of boron at grain boundaries is evaluated to be several % at a temperature of 950 °C, near A_{C3} , while the boron concentration inside the grain is substantially unaffected, Fig.11.

We think that boron retards the diffusive α/γ transformation during heating, because the grain boundary segregation of boron reduces grain boundary energy and makes the boundaries less

effective as heterogeneous nucleation sites for γ phase. This indicates that boron retards the diffusive α/γ transformation during heating. On the other hand, the nucleation of γ phase by

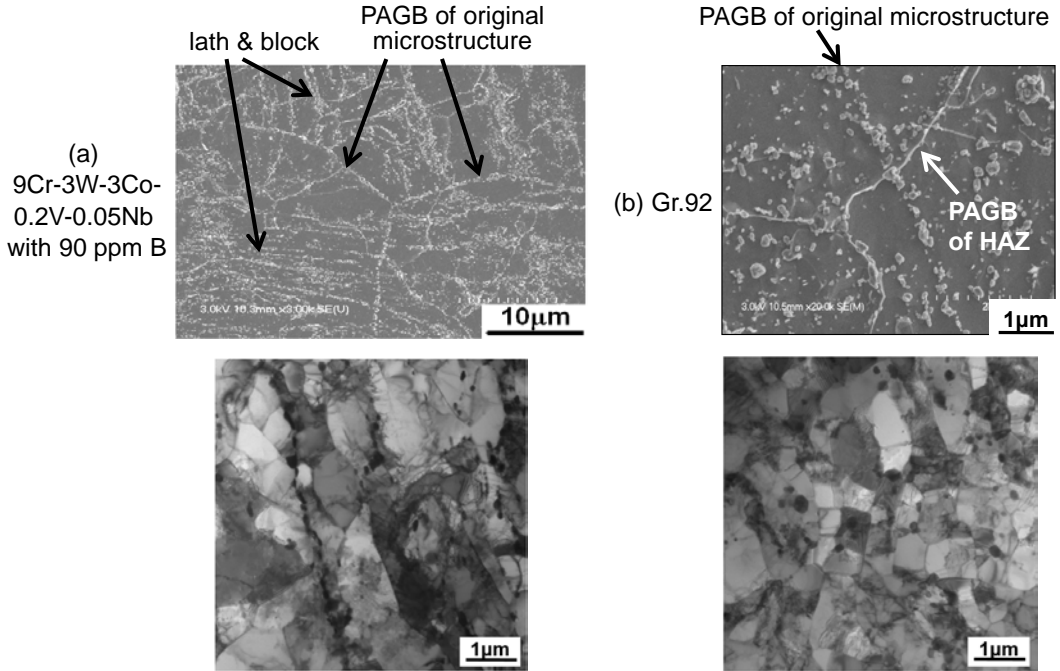


Fig.10 SEM and TEM micrographs of (a) 9Cr-3W-3Co-0.2V-0.05Nb steel with 90 ppm B and (b) Gr.92, after thermal cycle to 950 °C (near A_{C3}) and subsequent PWHT.

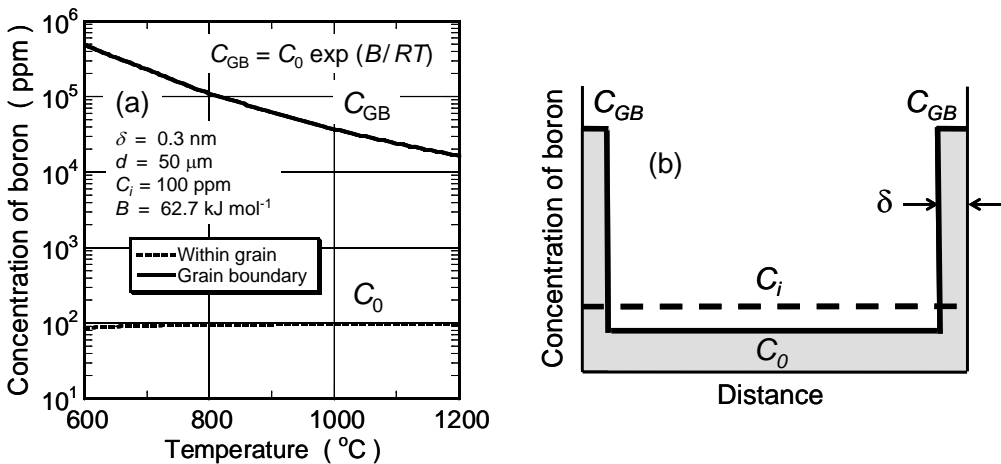


Fig.11 (a) Concentration of boron at grain boundaries and inside grain as a function of temperature (b) boron concentration profile.

diffusive transformation is promoted at GBs, because the presence of strained regions of lattice at GBs enable nuclei to be formed with a much smaller free energy of activation than that of nuclei inside the grain. The present results of the formation of fine grains at GBs of coarse grains in the

9Cr-boron steel suggest that driving force for diffusive α/γ transformation during heating is a little bit larger near GBs than the retardation effect by boron, Fig.12(a). The martensitic α/γ transformation inside the grain suggests that the retardation effect of diffusive α/γ transformation by boron is larger inside the grain than the driving force for diffusive α/γ transformation, Fig.12(a). The soluble boron concentration in the matrix is estimated to be about 20 ppm as shown in Fig.12(b), by assuming the initial boron concentration is 100 ppm, the enriched boron concentration in $M_{23}C_6$ carbides is 0.5% (500 ppm) as can be seen from Fig.3, and the mole fraction of $M_{23}C_6$ carbides is 0.0016 by thermo-calc. We think that the soluble boron concentration of 20 ppm in the matrix can retard the diffusive α/γ transformation and hence the martensitic α/γ transformation takes place inside the grain during heating.

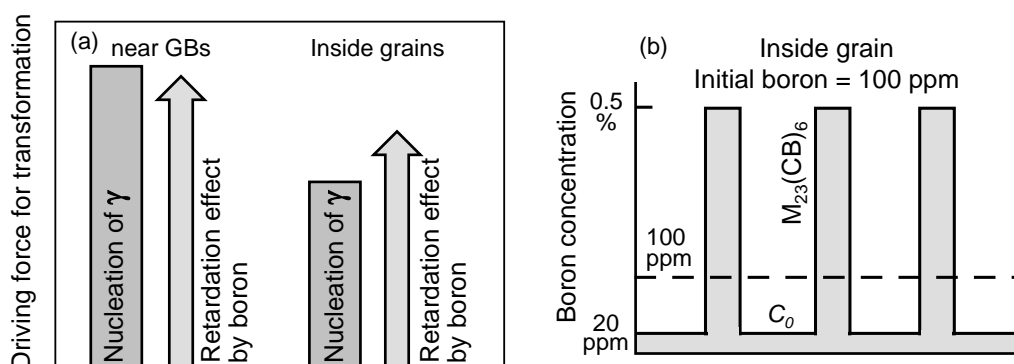


Fig.12 (a) Schematic drawing of driving force for α/γ transformation during heating and (b) boron concentration distribution inside grain.

Alloy design of MARBN : high strength and no Type IV fracture

Based on the suppression of Type IV fracture using boron and the improvement of long-term creep strength of base metal using both boron and fine MX nitrides without any formation of BN during normalizing, a 9Cr-3W-3Co-0.2V-0.05Nb steel containing 120-150 ppm boron and 60-90 ppm nitrogen was alloy-designed. The steel is denoted MARBN, which means MARtensitic 9Cr steel strengthened by Boron and fine MX Nitrides. As already described, the addition of boron as low as 40 ppm is enough to suppress the Type IV cracking in welded joints (Fig.8). However, the maximum creep strength of welded joints is limited by the creep strength of base metal and the increase in boron concentration improves long-term stability of base metal (Fig.1). Therefore, the addition of boron higher than 100 ppm is recommended. At a boron concentration of 120-150 ppm, the nitrogen concentration should be less than 100 ppm to avoid the formation of BN at normalizing temperature. The optimum nitrogen concentration is 60-90 ppm from a point of view of creep strength (Fig.4).

Figure 13 shows the creep rupture data for the base metal and welded joints of MARBN at 650 °C, together with those for the 9Cr-3W-3Co-0.2V-0.05Nb steel containing 139 ppm boron and 34 ppm nitrogen (Fig.1) and P92. MARBN exhibits a little bite higher creep rupture strength of base metal than the 9Cr steel containing 139 ppm boron and 34 ppm nitrogen, which results from the nitrogen effect. It should be noted that MARBN exhibits not only much higher creep rupture strength of base metal than P92 but also substantially no degradation in creep rupture strength of welded joints compared with the base metal.

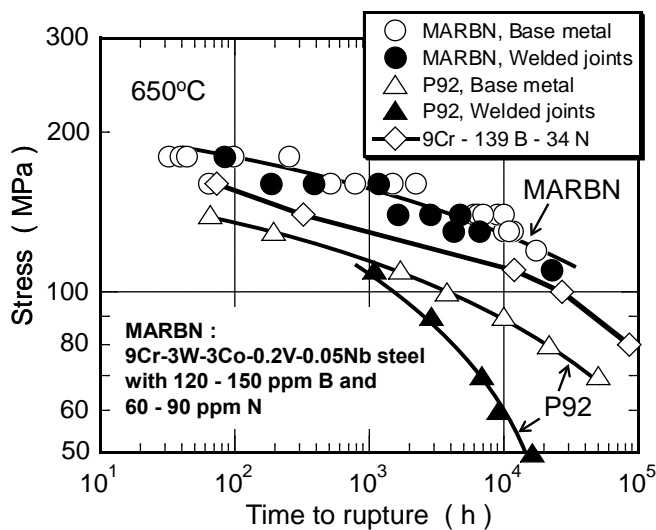


Fig.13 Creep rupture data for base metal and welded joints of MARBN, P92 and 9Cr-3W-3Co-0.2V-0.05Nb steel containing 139 ppm boron and 34 ppm nitrogen at 650 °C.

Improvement of oxidation resistance by pre-treatment

The formation of protective Cr-rich oxide scale is achieved by several pre-treatments, such as the pre-oxidation treatment in argon gas at 600 to 700 °C [14-15]. The long-term stability of Cr₂O₃ scale formed by the pre-oxidation treatment has been examined for the three steels, MARB1 with 100 ppm boron, MARB2 with 200 ppm boron and MARN with 0.05 % nitrogen but no addition of carbon in steam at 650°C, after the pre-oxidation treatment in Ar gas at 700 °C for 50 h, Fig.14. The formation of thin scale of Cr-rich oxides, having a thickness of less than 0.1 μm, is achieved by the pre-oxidation treatment. For the three steels MARB1, MARB2

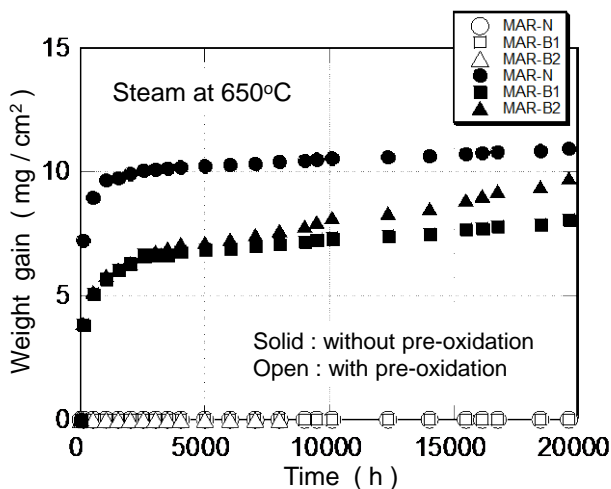


Fig.14 Weight gain of the steels in steam at 650 °C. MARB1 and MARB2: 9Cr-3W-3Co-0.2V-0.05Nb with 100 and 200 ppm boron, respectively, MARN : Low-carbon of 30 ppm C.

and MARN, the oxidation resistance in steam is significantly improved by the pre-oxidation treatment. The thin scale of Cr-rich oxides is stable during oxidation test in steam at 650 °C for a long time exceeding 20,000 h.

Comprehensive test program on the 9Cr-boron steel for getting approval is now being promoted by Nippon Steel & Sumitomo Metal Corporation (NSSC), on the base of cooperation between NSSC and some universities and institutes in Japan. Test results are presented in this conference by a companion paper [18].

Conclusions

- 1) The addition of boron and nitrogen to 9Cr-3W-3Co-VNb steel without any formation of BN during normalizing heat treatment improves the long-term creep strength of base metal and suppresses the Type IV cracking in HAZ of welded joints at 650 °C. The addition of boron also improves the creep rupture ductility.
- 2) The addition of excess nitrogen of 300 and 650 ppm to the 9Cr steel with 140 ppm boron causes the formation of large amount of BN during normalizing, which significantly reduces soluble boron and accelerates the degradation in creep rupture ductility.
- 3) The enrichment of soluble boron near PAGBs by the segregation is essential for the reduction of coarsening rate of $M_{23}C_6$ carbides near PAGBs, which enhances boundary and subboundary hardening near PAGBs, and also for the change in α/γ transformation behavior in HAZ of welded joints during heating, which produces approximately the same microstructure between HAZ and base metal. The enhancement of boundary and subboundary hardening by fine distributions of $M_{23}C_6$ carbides along boundaries retards the onset of acceleration creep, which effectively decreases the minimum creep rate and increases the creep life.
- 4) 9Cr-3W-3Co-0.2V-0.05Nb steel with 120-150 ppm boron and 60-90 ppm nitrogen (MARBN) exhibits not only much higher creep rupture strength of base metal than P92 but also substantially no degradation in creep strength due to Type IV fracture in HAZ of welded joints at 650 °C.
- 5) The formation of protective Cr-rich scale is achieved on the surface of 9Cr steel by pre-oxidation treatment in argon gas at 600 - 700 °C. This significantly improves the oxidation resistance of 9Cr steel in steam at 650 °C.

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